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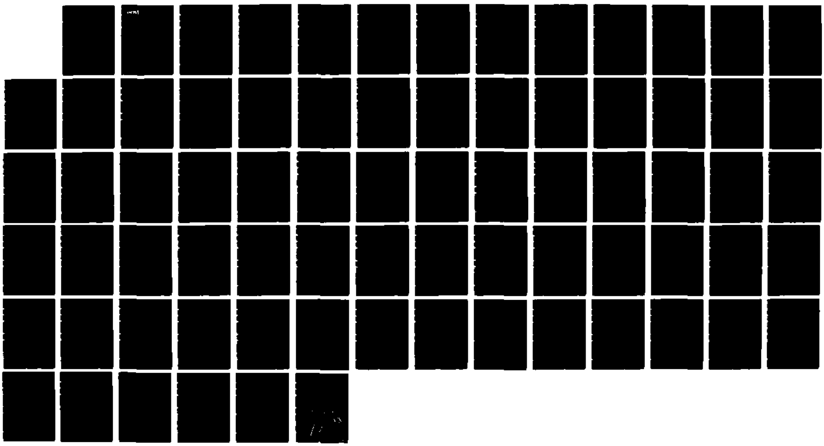

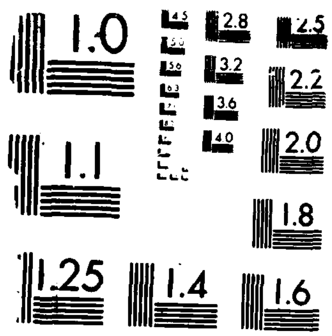

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Quality Assurance/Quality Control in Waste Site Characterization and Remedial Action

Final Report

M. P. Maskarinec
S. K. Holladay

Supported by

U.S. Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground, Maryland 21010-5401

Project Officer: Mary Ann Ryan

OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
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19 ABSTRACT *(Continue on reverse if necessary and identify by block number)*

In this report, efforts to advance the state of the art of Quality Assurance/Quality Control in waste site characterization and remedial action are detailed. The two most widely used and accepted QA/QC programs, the USEPA CLP and the USATHAMA IR QA plan, are compared and recommendations are provided for unification. The findings of a Working Group convened to discuss these issues are reported and the formation of a Task Force on Quality Assurance/Quality Control is announced.

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Quality Assurance/Quality Control in Waste Site Characterization and Remedial Action

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EXECUTIVE SUMMARY

This report details efforts to date to advance the state of the art of Quality Assurance/Quality Control (QA/QC) in waste site characterization and remedial action. The report is organized into three sections. The first section provides recommendations on the unification of the two widely used and accepted QA/QC programs: the U.S Environmental Protection Agency Contract Laboratory Program (USEPA CLP) and the U.S. Army Toxic and Hazardous Materials Agency Installation Restoration Quality Assurance (USATHAMA IR QA) plan. The second section compares the two plans in detail with the Guidelines given by the USEPA. The third section announces the formation of a Task force on Quality Assurance/Quality Control and reports on the findings of a Working Group convened to address these issues.

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UNIFICATION OF THE USATHAMA IR QA PLAN WITH THE USEPA CONTRACT LABORATORY PROGRAM

There currently exists a widespread agreement on the need for remedial action at past waste disposal sites. The approach usually taken is to study records pertaining to the site (preliminary investigation), to follow the investigation with a survey of contamination, and then to decide on a remedial action plan. Privately used sites are regulated under the Superfund Amendment and Reauthorization Act (SARA) by the USEPA. Sites used by government agencies, such as DOD and DOE, while regulated under SARA, are not generally cleaned up using SARA funds. An important aspect of the entire process is the analysis of large numbers of samples. Because of the increased emphasis on analytical methodologies, and the associated cost, it is crucial to ensure that the data produced be of acceptable quality. Therefore, strict Quality Assurance (QA) measures must be applied. Several different approaches to the various aspects of QA have been developed over the last decade, with perhaps the best known being the approach used by the USEPA under the Contract Laboratory Program (CLP). In addition, the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) has developed a QA plan to serve the needs of the Army Installation Restoration program. Both systems have been in existence for several years, and substantial experience has been gained. Because of the obvious similarity in the objectives of the two systems, this work was performed in an attempt to draw the two plans together. This was done in the following manner. A workshop was held to bring all interested parties together and discuss differences and similarities. Then, a detailed comparison was made of the two plans. Finally, this report was written to document the results of these efforts.

It must be noted that certain differences exist in the two programs which result from philosophical and logistical considerations beyond the issue of QA. Virtually all data generated in the CLP may eventually be called into court as evidence for prosecution or cost recovery actions. Therefore, it is necessary that the data be of courtroom quality. In the case of the IR plan, use of the data is generally restricted to direction of the remedial action phase. While not removing the requirement for high quality data, this end use does not mandate the degree of documentation used in the CLP. More important in the IR plan is streamlining of the data flow, and rapid identification of QA problems. In addition, the CLP has a relatively large number of participating laboratories compared to the IR system. This, combined with the end use difference, results in a need for more rigid standardization of the entire QA process. Interlaboratory comparability becomes much more crucial to the CLP than to the IR program. A related logistical difference is the fact that in the CLP, samples are collected and sent to a sample management facility, either central or regional. The samples are then distributed to the analytical facilities. In the USATHAMA case, samples are collected by

the prime contractor for the remedial action and either analyzed in house or sent to a subcontracting analytical facility. The CLP can therefore do a more effective job of providing blind QA samples (spikes, splits, or blanks) than can the IR plan. Taken together, these differences result in a marked reliance on external QA in the CLP, and a corresponding reliance on internal QA in the IR plan. Given this difference, it would appear that the two systems are not mutually exclusive, and that reconciliation of the programs might result in an even stronger unified plan.

The first issue to be resolved is the method of assuring initial laboratory proficiency. In the CLP, this is done by analysis of a performance evaluation sample. In the IR plan, the laboratory performs a certification study, which is used to establish the QA paramaters for the method. While not specifically required by the CLP, some type of initial certification must certainly be done by the laboratory in order to gain familiarity with the method prior to running the PE sample. It would seem prudent to establish guidelines for the certification process which allow the laboratory to prove competence in the method. **The certification procedure used by USATHAMA should be recommended or even required by the CLP.**

The use of performance evaluation samples has advantages and disadvantages. PE samples can rarely be provided which are truly blind. There is no assurance that the successful analysis of a PE sample reflects everyday laboratory performance. Furthermore, the analysis of PE samples is restricted to a relatively low frequency (quarterly) so that if problems are identified, large gaps exist in which laboratory performance is in question. Finally, the time required to analyze the PE data further increases the lag time. On the other hand, the PE sample is the only truly external check on laboratory performance, and the only means by which laboratories can be compared and rated. **Therefore, it is recommended that USATHAMA adopt the performance evaluation system used by the CLP, and that efforts be made to rapidly evaluate that data and report problems to the laboratory.**

Several differences exist between the two plans with respect to sampling and analysis. The only fundamental difference is that the CLP requires the analysis of all samples for compounds on the Hazardous Substances List (HSL) while the Army has contamination from military-specific compounds which do not appear on the HSL. When the IR plan is used for analysis of HSL compounds, the CLP methodology is followed. Therefore, the IR plan is equivalent from an analytical standpoint to the CLP, but includes in addition the QA required for non-CLP methods. Other differences include container cleaning procedures and holding times. Differences of this type can be handled experimentally, by performing an equivalency test. The IR plan does not require chain-of-custody procedures to be followed, unless the data is to be used in litigation. When used, these procedures are functionally identical to

those used in the CLP plan. Chain-of-custody procedures are a necessary part of good laboratory practice, and should always be used. **It is recommended that USATHAMA require CLP chain-of-custody procedures be followed for all samples.**

In terms of data management and communication, USATHAMA has developed a sophisticated computer-based system. All data is entered by the analyst into a personal computer, checked for completeness, and transferred to a mainframe. The laboratory is required to submit all raw data at the end of the contract. In the CLP, all data generated pertaining to a particular sample is submitted with the results from that sample in a data package. This is clearly an example of differences resulting from the end-use situation mentioned previously. The USATHAMA system is far more workable from the standpoint of remedial action decision making, but the CLP system is required for litigation. However, it must be pointed out that the data in either case is available, and that nothing has been lost. Therefore, it should be possible for the IR software to produce a CLP data package on demand. If this can be done, then there is no practical reason to change either plan. **It is recommended that USATHAMA demonstrate the ability to produce a CLP data package.**

The software package used in the IR plan has additional features which are quite desirable from a QA standpoint, including the ability to generate QC charts. While it is implied in the CLP that QC charts should be kept, no formal requirement exists and no standardized approach is provided. QC charting has several advantages: rapid identification of out-of-control situations, assurance that performance is consistent on a day-to-day basis, and documentation that the laboratory is performing well on each and every sample. Thus, QC charting can serve as an adjunct to the PE system, and alleviate the drawbacks of PE samples. The question is: what should be charted? Since the surrogates and internal standards used in the CLP are present in every sample, it seems logical to require that the surrogate recoveries and internal standard areas be control charted. **It is recommended that the CLP use the USATHAMA software package and require control charts for surrogate recoveries and internal standard areas. It is further recommended that USATHAMA provide USEPA with the software and documentation.**

A difference also exists in the area of matrix spiking. The CLP requires a matrix spike and matrix spike duplicate to be run for each matrix. The IR plan uses a standard matrix. The CLP matrix spike does provide additional information on the performance of the methods with respect to individual matrices. However, it can be difficult to determine when one matrix differs from the previous one. On the other hand, the IR method provides a historical record of the performance of the method with time. Given that the surrogates are present in every sample and can be considered matrix spikes, the issue seems to be whether any additional information can be obtained from sample matrix

spikes. Furthermore, the issue of interlaboratory comparability - so important to the CLP program - would be better served by the use of a standard matrix than by use of sample matrices. **It is recommended that the CLP drop the requirement for sample matrix spikes and matrix spike duplicates and adopt the standard matrix approach used by USATHAMA.**

One of the major problems faced by analytical laboratories doing work in the remedial action area is the audit. Each contracting agency has its own style of auditing, and preparation for the audit depends on the needs and requirements of the auditing agency. In both the CLP and the IR programs, the audit is used as a tool to improve the performance of the laboratory. Because of all of the differences listed previously, the audits take on a different flavor depending on which agency is auditing. However, if the modifications recommended in this document can all be made, the audit could be performed by either USATHAMA or CLP personnel and would suit the needs of both programs. This would result in substantial savings to the agencies involved and would be very convenient for the laboratories.

In summary, the recommendations made here are the result of an objective comparison of the CLP and the USATHAMA IR QA plan. These recommendations are made with the goal of improving quality assurance and quality control in environmental measurements related to waste site characterization and remedial action. An additional goal is the reduction of the cost associated with QA. Two approaches are feasible in this regard. The most easily adopted from the philosophical viewpoint is the declaration of equivalency of the two plans. To this end, a detailed comparison follows of the two plans with the general guidelines set forth in the USEPA sixteen point QA project plan. While this would be expedient, the separate-but-equal approach is far less desirable than the approach of combining the best of both. To that end, continuing communication between the principal agencies and a willingness to cooperate on these issues is mandatory. **It is recommended that the USEPA grant equivalency to the USATHAMA IR QA plan, but at the same time strive for unification.**

REVIEW OF THE USATHAMA QA PROGRAM (MARCH, 1987) AND THE USEPA CONTRACT LABORATORY PROGRAM USING THE USEPA SIXTEEN-POINT QA PROJECT PLAN

1. Title Page with Provision for Approval Signatures

2. Table of Contents

3. Project Description

4. Project Organization and Responsibility

A diagram of the lines of communication for USATHAMA IR projects (USATHAMA QA Program, 2nd edition, March, 1987) has been included in Appendix A and a diagram of the program principals of the USEPA (User's Guide to the Contract Laboratory Program, October, 1984) in Appendix B.

5 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability

Laboratory Certification

USATHAMA	EPA-CLP	COMMENTS
Contract award	Laboratory selection	USATHAMA specifies that the QA/QC plan must be documented and in practice before samples arrive. EPA-CLP requires a documented QA/QC SOP, but does not specify a timetable.
Development of Project QC Plan A statement of adherence or reference to the USATHAMA QA Program A detailed account of how the QA Program will be implemented A description of the organization, responsibilities, and decision-making authorities of the contractor project team A description of sampling team and analyst training in technical skills, standard QC, and essential elements of QA Program Procedures for sampling, preservation, and shipment of samples Sample inspection and lot sizing Instrument calibration Logs (field, instrument, sample, QC) Analytical reference materials Procedures for verifying and documenting the quality of lab water Control charts Methods and criteria for determining when sampling or analytical systems are out of control, including holding times Actions to be taken to correct out-of-control situations, and how actions will be reported and documented A list of personnel responsible for data review and sequence of review prior to submittal	Development of written QA/QC standard operating procedure (SOP) containing these essential elements: Organization and Personnel QA policy and objectives QA organization Personnel training Document control and revisions Facilities and Equipment Procurement and inventory procedures Preventive maintenance Analytical Methodology Calibration and operating procedures Sample Custody Quality Control Quality control procedures Control checks and internal audits Reference material analysis Blank analysis Matrix spike and matrix spike duplicate analysis Internal audits Data Handling Data handling, reporting, and record-keeping procedures Data validation	

5 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA CLP	COMMENTS
Generation and submission of pre-certification performance data package	Preparation for performance evaluation samples	EPA CLP requires the analysis of "blind" evaluation samples for evaluation of laboratory performance USATHAMA requires analysis of certification samples which are not blind
Precertification method description (preparation and analysis of standards)		
Standardized method written to be laboratory-specific		
Development of method	Tuning and GC/MS mass calibration	Assumption is made that GC/MS tuning is described as part of the method
Submit documentation for proposed method		
Analytical procedures testing		
Documentation of method in standard format		
Generation of performance data packages		
Review by USATHAMA Analytical Branch		
Assignment of method number after final approval		
Precertification calibration data		
Construction of calibration curve		
Prepare and analyze each standard in duplicate to bracket desired range for certification TRL = Target Reporting Limit (designated by USATHAMA)	Construction of Calibration Curves	
Class 1	Pesticides	
Blank, 0.5, 1, 2, 5, and 10 times the TRL plus expanded range	Established retention time windows Run evaluation standard mix at three concentrations Run standard mix of pesticides Run individual Aroclors	
Class 1A and Class 1B		

5. QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA-CLP	COMMENTS
Blank, 0.5, 2, and 10 times the TRL + range extension (10% for inorganic and 25% for all others)	GCMS: semivolatiles and volatiles require 5 point calibration curve with specified concentrations of 20, 50, 80, 120, and 160 ngs	
Tabulate and graph response vs. concentration Lack of fit (LOF) Zero intercept (ZI)	Tabulation of calibration data GCMS: Relative response factors Relative standard deviation Calibration factors GC: % RSD % Breakdown	
Certified calibration check standard - Class 1 and 1B only	Verification of performance checks GCMS: System performance check Calibration check	
Class 1		
Two calibration check standards should be analyzed, one at the beginning and one at the end of the day - near high end of range	GC: Retention time shifts % Breakdown	
Class 1B		
One calibration check standard should be analyzed at the beginning of the day. New high end of range		
Results of identification and purity analyses for all off-the-shelf reference materials		
Checklist completed by the QAC		
Approval by USATHAMA of Pre-certification Performance Data Package and Project QA Plan		
Generation and submission of Certification Performance Data Package Final USATHAMA-approved copy of the Precertification Performance		

5. QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA-CLP	COMMENTS
Data Package		
Total method description in USATHAMA format containing approved deviations in the standardized method and laboratory-specific information concerning conduct of the method	Submission of Standard Operating Procedures	
Initial calibration MTR = minimum testing range TRL = target reporting limit * = times		Calibration procedure for semivolatiles and volatiles for EPA-CLP resembles USATHAMA Class 1 more than Class 1A which is reserved for all GC/MS methods. However, USATHAMA calibration for pesticides (assuming Class 1) is more stringent than *EPA-CLP*.
Class 1 MTR; blank, 0.5, 1, 2, 5, *10, and *10TRL 7 standards + 2 check standards MTR + 1 range extension; 10 standards + 2 check standards (20, 50, 100, 100) MTR + 2 range extensions; 13 standards + 2 check standards (20, 50, 100, 200, 500, 1000, 1000)		
Class 1A MTR; blank, 0.5, 2, 10 and 10 *TL*; 5 standards MTR + 1 range extension; (50, 200, 200); 7 standards MTR + 2 range extension; (50, 200, 500, 2000, 2000); 9 standards	*GCMS*: *semivolatiles* and volatiles require a 5 point initial calibration at specified concentrations	
Class 1B - same as 1A plus 1 check standard		
Class 2 - 6 standards, blank, and 1 triplicate TRL		
Daily calibration		Daily calibration for USATHAMA requires analysis of a high standard twice whereas EPA-CLP requires analysis of a lower range standard.
Class 1/Class 1A/Class 1B 2 standards for MTR - *10 and *10 TRL 2 standards for MTR + 1 range	GCMS: 50 total ngs, standard analyzed each 12 hours	

5. QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA-CLP	COMMENTS
extension - *100 and *100 TRL 2 standards for MTR and 2 range extensions - *1000 and *1000 TRL	Response must be within 25% for organics of mean response of 5 initial calibration standards	
Class 2 4 standards (MTR) and blank and 1 TRL (duplicate)		
Certification samples (prepared in standard matrix)	Performance Evaluation	With USATHAMA certification samples, the participating laboratory knows immediately whether problems exist in sample preparation and/or analysis. However, this same knowledge is available to EPA-CLP laboratories only if the results of the evaluation samples are returned promptly.
Class 1/Class 1B MTR: 24 Blank, 0.5, 1, 2, 5, and 10 TRL (4 consecutive days)	*Samples prepared by EMSL/LV are sent to* laboratory	
MTR + 1 range extension: 36 Blank, 0.5, 1, 2, 5, 10, 20, 50, 100 TRL (4 days)		
MTR + 2 range extentions: 48 Blank, 0.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, and 1000 TRL (4 days)		
Class 1A MTR: 8 Blank, 0.5, 2, and 10 TRL (duplicate)		
MTR + 1 range extensions: 12 Blank, 0.5, 2, 10, 50, and 200 TRL (duplicate)		
MTR + 2 range extensions: 16 Blank, 0.5, 2, 10, 50, 200, 500, and 2000 TRL (duplicate)		
Class 2 MTR: 8 Blank, 1 TRL (quadruplicate)		
Statistical Analysis of the Data	Data Package	
Tabulation of found vs. target concentration	Sample Traffic Report Sample Data Summary Package	
LOF and ZI test calculations and	Case narrative	

5. QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA-CLP	COMMENTS
results for the pooled data set for found vs. target concentration	Target compound results-(Form I) Tentatively identified compounds-(Form I)	
Linear regression Confidence bounds Reporting limit Accuracy Standard deviation Percent Imprecision (% RSD) Percent Inaccuracy	Surrogate spike analysis results-(Form II) Matrix spike/matrix spike duplicate-(Form III) Blank data-(Form IV and Form I)	EPA-CLP surrogate spike is a measure of percent inaccuracy and matrix spike/matrix spike duplicate is a measure of percent imprecision.
Narrative evaluation of effectiveness of method Checklist completed by QAC	Sample Data Package Case narrative Traffic reports Volatiles data QC summary Surrogate spike results, Form II Matrix spike results, Form III Method blank summary, For GC/MS tuning standard, Form V Sample data TCL results, Form I Ion chromatograms Mass Spectra Library search spectra for TIC Quantitation of TIC Manual work sheets Standards data Initial calibratin data, Form VI Continuing calibration data, Form VII Internal standards summary, Form VIII Raw QC data BFB mass spectra Blank data, Form I Ion chromatograms Mass spectra Library search spectra for TIC Quantitation of TIC Manual work sheets Matrix spike results, Form I Matrix spike duplicate results, Form I Semivolatiles data QC summary	

5. QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA-CLP	COMMENTS
Calibration data, tabulation of concentration vs. response (a) Initial (b) Daily	Surrogate spike results, Form II Matrix spike results, Form III Method blank summary, Form IV	
Calibration curves (instrument response on ordinate vs. concentration on abscissa) Data demonstrating that the response for Daily Calibration standards was within required percentage of highest standard Copies of chromatograms for high and low standards - certification samples analysis date and time target concentration test name reference to calibration curve reference to analytical logbook each peak labeled Spectra for all target analytes	GC/MS tuning standard, Form V Sample data TCL results, Form I Ion chromatograms Mass spectra Library search spectra for TIC Quantitation of TIC Manual work sheets Standards data Initial calibration data, Form VI Continuing calibration data, Form VII Internal standards summary, VIII Raw QC data BFB mass spectra Blank data, Form I Ion chromatogram Mass spectra Library search spectra for TIC Quantitation of TIC Manual work sheets Matrix spike results, Form I Matrix spike duplicate results, Form I Pesticides/PCB data QC summary Surrogate spike results, Form II Matrix spike results, Form III Method blank summary, Form IV Sample data TCL results, Form I Gas chromatograms Confirmation gas chromatograms Manual work sheets GPC chromatograms GC/MS raw spectra Standards data Evaluation standards summary, Form VIII	

5. QA Objectives for Measurement Data in Terms of Precision, Accuracy, Completeness, Representativeness, and Comparability (cont.)

USATHAMA	EPA-CLP	COMMENTS
	Standards summary, Form IX Identification, Form X Chromatograms Raw QC data Blank data, Form I Gas chromatograms Matrix spike results, Form I Gas chromatograms and printouts Matrix spike duplicate results, Form I Gas chromatograms and printouts	
	Data evaluated for accuracy by NPO (National Program Office) and audited by EMSL/LV personnel	
	Quality control data goes into EMSL/LV database for trend analyses, etc. On-site laboratory evaluation	

6. Sampling Procedure

(A) Sample containers (Appendix C and Appendix D)

Inorganics

	USATHAMA	EPA-CLP	COMMENTS
Water	Polyethylene (Exception: glass bottle and top for dissolved oxygen)	Polyethylene (Medium level requires wide-mouth glass jar)	
Soil	Amber glass bottle with Teflon-lined cap	Wide-mouth glass jar	

Organics

	USATHAMA	EPA-CLP	COMMENTS
Water			
Volatiles	Glass vial with Teflon-lined septum cap	Glass vial	
Semivolatiles	Amber glass bottle with Teflon-lined cap	Amber glass bottle (wide-mouth glass jar for medium level)	
Soil			
Volatiles	Glass vial with Teflon-lined septum cap	Wide-mouth glass vial	
Semivolatiles	Amber glass bottle with Teflon-lined cap	Wide-mouth glass jar	

6. Sampling Procedure (cont.)

(B) Sample container cleaning procedures (Appendix E and Appendix F)

	USATHAMA	EPA-CLP	COMMENTS*
Polyethylene bottles and caps	5% sodium hydroxide deionized water 5% Ultrex nitric acid/water deionized water air dry	Cleaning procedure used by EPA-CLP Sample Bottle Repository not known at this time.	These procedures are referenced by companies advertising precleaned bottles Polyethylene bottles detergent tap water 1:1 nitric acid tap water 1:1 hydrochloric acid tap water distilled water VOA vials detergent tap water distilled water dry at 105°C EXTRACTABLE bottles detergent tap water distilled water acetone hexane (pesticide quality) air dry (muffle furnace heating may be substituted for solvent rinses)
Amber glass bottles or vials	detergent distilled water acetone methylene chloride hexane air dry heat to 200°C		
Bottle caps	remove paper liners detergent distilled water dry at 40°C		
Teflon liners	detergent distilled water acetone hexane air dry place liners in cleaned caps heat to 40°C for 2 hours cool		detergent tap water distilled water dry at 105°C for 1 hour

*EPA 40 CFR 136 "Guidelines for Establishing Test Procedures for the Analysis of Pollutants"

6. Sampling Procedure (cont.)
(C) Sample holding times (Appendix C and Appendix D)

Definitions

USATHAMA - maximum time allowable between sample collection and analysis

EPA-CLP - maximum time allowable between verified time of receipt (VTSR) and analysis

	USATHAMA	EPA-CLP	COMMENTS
Inorganics			
Metals	6 months (chromium VI-24 hours)	6 months	Major difference in definition of holding time probably reflects also the differences noted in the holding times. Holding times are selected arbitrarily and by convenience. Neither plan is necessarily correct.
Mercury	28 days	26 days	
Cyanide	14 days	14 days	
Organics			
Extractables			
Soil	7 days	10 days	
Water	7 days	5 days	
Volatiles			
Soil	14 days	10 days	
Water	14 days (with pH adjustment) 7 days (no pH adjustment)	10 days	

6 Sampling Procedure (cont.)

(D) Sample preservation and storage conditions

Definition of sample storage termination:

USATHAMA - sample storage shall only be terminated after all analytical results have been validated to Level 3 in the USATHAMA Data Management System

EPA-CLP - sample extracts shall be retained for 365 days after data submission

	USATHAMA	EPA-CLP	COMMENTS
Inorganics			The length of the sample archival depends on the program.
Metals*			Sample preservation and storage conditions are basically the same except for volatiles. Preservation in USATHAMA plan is consistent with EPA 40 CFR 136.
Soil	Cool, 4°C		
Water	HNO_3 to pH<2 (except chromium VI)	HNO_3 to pH<2	
Mercury			
Soil	Cool, 4°C		
Water	HNO_3 to pH<2	HNO_3 to pH<2	
Cyanide			
Soil	Cool, 4°C		
Water	Cool, 4°C NaOH to pH>12 0.6g ascorbic acid	Cool, 4°C NaOH to pH>12 0.6g ascorbic acid	
Organics			
Extractables	Store in dark Cool, 4°C	Store in dark Cool, 4°C	
Volatiles	Cool, 4°C 0.008% $Na_2S_2O_3$ if chlorine is present HCl to pH<2 (or analyze within 7 days)	Store in dark Cool, 4°C	

*Samples should be filtered before adding preservative for dissolved metals.

7. Sample Custody

USATHAMA	EPA-CLP	COMMENTS
<u>Field sampling</u>		
Sample acquisition as well as distinguishing information recorded in bound logbook	Sample Traffic Report	
Sample label Installation name Unique sequential field sample no. Sampling date Analytes of interest Preservative/filtration	Sample tag-information defined by EPA National Enforcement Investigations Center (NEIC) CLP case/SAS no(s). — CLP sample no. Project code — Station no. Date — Time Station location — Samplers Remarks — Tag no. Lab sample no.	
Use of formal Chain-of-Custody procedures implied for litigation	Chain-of-Custody Record	USATHAMA needs to document chain-of-custody procedures.
<u>Laboratory Operation</u>		
Sample login		USATHAMA's procedures for sample login and analysis are covered in the Project QC Plan under Laboratory Certification.
Samples are logged into a project-specific logbook	Standard operating procedures required for: receipt of samples; maintenance of custody; sample storage	
Samples are grouped into analytical lots, ordered and assigned a USATHAMA sample identification number (QC samples also)		
Sample analysis		
Bound logbooks required for: reference materials; operational activities which occur during sample handling; instrument operation	Standard operating procedure for tracking the analyses of samples required Bound logbooks required for entering all observations and results not on pre-printed data sheets	
<u>Document control</u>		
All documentation shall be in ink	Standard operating procedure for the assembly of completed data	USATHAMA requires a list of personnel responsible for data review and sequence of review in Project QC Plan.
Errors shall be corrected by crossing a line through the error, entering the correct information, and dating and initialing the change	All documentation shall be in ink	

7. Sample Custody (cont.)

USATHAMA	EPA-CLP	COMMENTS
Computerized logging systems may not be used for original records	Errors shall be corrected by crossing a line through the error, entering the correct information, and dating and initialing the change	
Logbook should be installation-specific	Documentation is cross-checked for consistency	
	Documents are numbered and inventoried	

8 Calibration Procedures and Frequency

USATHAMA	EPA CLP	COMMENTS
<u>Initial calibration</u>		
Frequency (a) 1st day of certification analyses (b) Instrumental start-up (not daily) (c) Analyzing different analytes (d) Daily calibration fails If samples are analyzed on the same day as initial calibration, one standard at the highest concentration must be analyzed after analyses are completed	Frequency Prior to analysis of samples and if daily calibration fails	Frequency requirements are equivalent
Concentration of standards Class 1 MTR; blank, 0.5, 1, 2, 5, *10, and *10 TRL, 7 standards + 2 check standards MTR + 1 range extension; 10 standards + 2 check standards (20, 50, 100, 100) MTR + 2 range extensions; 13 standards + 2 check standards (20, 50, 100, 200, 500, 1000, 1000) Class 1A MTR; blank, 0.5, 2, 10, & 10 TRL; 5 standards MTR + 1 range ext.; (50, 200, 200); 7 standards MTR + 2 range ext.; (50, 200, 500, 2000, 2000); 9 standards Class 1B - same as 1A plus 1 check standard Class 2 - 6 standards, blank, and 1 triplicate TRL	Concentration of standards GC/MS (≈ Class 1A) Volatiles 20, 50, 100, 150, and 200 µg/L The % RSD for each calibration check compound must be less than or equal to 30.0% The minimum acceptable average relative response factor is 0.300, 0.250 for bromoform Semivolatiles 20, 50, 80, 120, and 160 total nanograms The % RSD for each calibration check compound must be less than or equal to 30.0% The minimum acceptable average relative response factor is 0.05	Calibration procedure for semivolatiles and volatiles for EPA-CLP resembles USATHAMA Class 1 more than Class 1A which is reserved for all GC/MS methods However, USATHAMA calibration for pesticides (assuming Class 1) is more stringent than EPA-CLP.
Certified check standards Class 1 - two stds - beginning & end of day Class 1B - one std - beginning of day - near high end of range If acceptability limits are exceeded, immediate reanalysis occurs, followed by a new initial calibration if necessary	GC (≈ Class 1) Pesticides Evaluation standard Mixture of aldrin, endrin, and 4,4,'-DDT at concentrations of 20%, 50%, and 100% full-scale Individual standard mixes and aroclors	Certified USATHAMA check standards made from the stock solution used during certification allow a continual check of laboratory performance.

8. Calibration Procedures and Frequency (cont.)

USATHAMA	EPA-CLP	COMMENTS
	The % RSD for evaluation standard mix compounds must be <10.0%. The % breakdown for endrin or 4,4'-DDT must not exceed 20.0%. The calibration factor for each individual standard must not exceed a 15.0% difference for a quantitation run nor exceed a 20.0% difference for a confirmation run.	
<u>Daily calibration</u> Class 1, 1A, 1B zero-intercept Highest concentration standard is analyzed at beginning and end of day. Response must be within 10% for inorganic and 25% for others of the mean response for the same concentration as determined for precertification and certification for the 1st 7 calibrations After 7 calibrations, response must agree within 2 standard deviations	GC/MS (= Class 1A) Volatiles 50 $\mu g/L$ standard is analyzed every 12 hours The % difference for each calibration check compound must be less than or equal to 25.0%. The minimum relative response factor for the system performance check compounds is 0.300 (0.250 for bromoform)	Daily calibration for USATHAMA requires analysis of the high standard twice whereas EPA CLP requires analysis of the lower range standard. The quality of data should be equivalent.
Corrective action Reanalyze daily standard Initial calibration repeated		
Non-linear or non-zero intercept Analyze low, middle, and high calibration standards at beginning of day and low and high standards at the end of the day. (If quadratic, four standards) Responses must fall within 2 std. deviations of the mean response	Semivolatile 50 total ngs standard is analyzed every 12 hours The minimum relative response factor for the system performance check compounds is 0.050 The % difference for each calibration check compound must be less than or equal to 25.0%	
Class 2 One blank and one calibration standard at the CRL analyzed at beginning and end of sample analysis		
	Pesticides Analyze evaluation standard Mix B and individual standard Mix A or B alternately after every 5 samples The % difference in retention time for the dibutylchlorendate must not exceed	

8. *Calibration Procedures and Frequency (cont.)*

USATHAMA	EPA-CLP	COMMENTS
	0.3% for capillary or 2.0% for packed column The % breakdown for 4,4'-DDT or endrin must not exceed 20.0%	
<u>Reference Materials</u>		USATHAMA provides reference materials to prepare all standard solutions.
Standard Analytical Reference materials traceable to NBS	EMSL/LV provides standard materials from its QA Materials Bank for performing initial instrument calibration and as reference standards	EPA also makes available QC samples intended for periodic (quarterly) use as independent checks on each laboratory's own QC activities.
Interim reference material (a) Central QA Lab (b) EPA (c) NBS		No practical difference
Off-the-shelf material (a) Positive identification (b) *Estimate of purity*		

9. Analytical Procedures

USATHAMA uses EPA standardized methods for commonly encountered analytes and USATHAMA-specific methods are used when no EPA comparison is available.

10 Data Reduction, Validation, and Reporting

USATHAMA	EPA-CLP	COMMENTS
<u>Data reporting</u>		Note difference in reporting of soil/sediment data. Either report is acceptable if the end user is aware of the difference.
CRL = certified reporting limit		
Data is not adjusted by any correction factors (such as accuracy, % moisture, and dilution factor), but is reported in the as-received condition	Soil/sediment data is adjusted to Dry Weight Basis	
Class 1, 1A, 1B All values less than CRL will be reported as <RL	Values less than quantitation limit are reported with J qualifier	
Number of Significant Figures to be used in Reporting Data		
Class 1 and 1B No dilution - 3 significant figures Dilution - 2 significant figures Noncertified analytes - retention time	GC/MS: Report data to 2 significant figures	
Class 1A No dilution - 2 significant figures After dilution - 1 significant figure Screening for noncertified - 1 significant figure	GC-Pesticides: Report data to 2 significant figures	
Class 2 CRL - 2 significant figures Reported as >, <, = CRL	<u>Deliverables</u> Inorganic (1) Weekly process reports (2) Sample traffic report (3) Sample data package Tabulated results Raw data Copies of logbook entries	
<u>Deliverables</u> Specific instructions for format, coding, and submission are provided in the IRDMS User's Guide	Organic (1) Narrative report (2) Sample traffic report (3) Quality control summary (4) Sample data (5) Raw sample data (6) Standards package (7) QC data package	

11. Internal Quality Control Checks

USATHAMA	EPA-CLP	COMMENTS
__Types__		USATHAMA does not require matrix spiking (as EPA perceives) for organics. Matrix spikes could easily be added to USATHAMA plan. Frequency should be as in CLP. Matrix spikes are probably not necessary if surrogates are added to each sample, unless surrogate recovery is low.
Class 1 and Class 1B Method blank Spikes of control analytes in standard matrices Class 1A (GC/MS) Method blank/surrogate spikes Surrogates spikes in every field sample Class 2 Method blank Spikes of control analytes in standard matrices	__Inorganics__ Preparation blank analysis Interference check sample analysis ICP serial dilution analysis Matrix spike analysis Duplicate sample analysis Furnace AA QC Analysis (Method of Standard Addition may be required under certain conditions) Laboratory quality control sample analysis __Organics__ Method blank analysis Surrogate spike analysis Matrix spike/Matrix spike duplicate analysis	
__Frequency per lot__		
Class 1 One - standard matrix method blank Three standard matrix spikes 2, 10, & 10 CRL Class 1A One - standard matrix method blank spike (surrogate, 10 CRL) All field samples spiked with surrogate - 10 CRL Class 1B One - standard matrix method blank One - standard matrix spikes - 10 CRL Class 2 (a) One - standard matrix method blank (b) One standard matrix spike - 1 CRL	__Inorganics__ Preparation blank - every 20 samples received or with each batch of samples digested whichever is more frequent Interference check sample - analyzed at beginning and end of each analysis run or a minimum of twice per 8 hour working shift ICP serial dilution - each group of samples of a similar matrix type and concentration for each case of samples or for each 20 samples received, whichever is more frequent Spiked sample and duplicate sample - at least one for each group of samples of a similar matrix and concentration for each case of samples or for each 20 samples received, whichever is more frequent	

11. Internal Quality Control Checks (cont.)

USATHAMA	EPA-CLP	COMMENTS
	Laboratory control sample - one for each group of 20 samples of a similar matrix or for each batch of samples digested whichever is more frequent	
	<u>Organics</u>	
	Method blank analysis Volatiles For the analysis of volatile TCL compounds, a method blank analysis must be performed once for each 12-hour time period during the analysis of samples from: o each case, OR o each 14 calendar day period during which samples in a case are received (said period beginning with the receipt of the first sample in that sample delivery group), OR o each 20 samples in a case that are of similar matrix (water or soil) or similar concentration (soil only), <u>whichever is most frequent</u>, on each GC/MS system used to analyze samples Extractables For the analysis of extractable TCL compounds, a method blank analysis must be performed once: o each case, OR o each 14 calendar day period during which samples in a case are received (said period beginning with the receipt of the first sample in that sample delivery group), OR o each 20 samples in a case that are of similar matrix (water or soil) or similar concentration (soil only), OR	Method blank requirements are equivalent.

11. Internal Quality Control Checks (cont.)

USATHAMA	EPA CLP	COMMENTS
	o whenever samples are extracted by the same procedure (separatory funnel or continuous extraction), whichever is most frequent, on each GC/MS or GC system used to analyze samples	
	Surrogate spike analysis All blanks, field samples, matrix spikes, and matrix spike duplicates will be spiked with surrogate compounds	
	Matrix spike analysis A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, once: o each case of field samples received, OR o each 20 field samples in a case, OR o each group of samples of a similar concentration level (soils only), OR o each 14 calendar day period during which samples in a case were received (said period beginning with the receipt of the first sample in that sample delivery group), whichever is most frequent.	See earlier comments on matrix spiking.

11 Internal Quality Control Checks (cont.)

USATHAMA	EPA-CLP	COMMENTS
<u>Preparation</u>		
Assigned sample number during logging-in process Spiked samples (excluding water samples) must be allowed to stand for one hour before continuing the analysis		
<u>Data Reporting</u>	<u>Data Reporting</u>	Method blanks can be corrected in USATHAMA plan, but cannot be *corrected in CLP. Blank correction* is fine, but any time this is done the value should be documented.
Class 1 Minimum of 3 significant figures Method blank: can be corrected - reported by concentration Control charts	*Reporting of quality control samples* handled just as samples are	CLP should require control charts at least for surrogates and internal standards. In earlier IFB's, control charts were required for internal standards.
Class 1A 2 significant figures Method blank: can be corrected - reported by concentration Control charts	Soil/sediment results are corrected for percent moisture and reported on a dry weight basis No corrections are made for method blanks	
Class 1B Minimum of 3 significant figures Method blank: can be corrected - Reported by concentration Control charts		
Class 2 Minimum of 2 significant figures No control charts		

12 Performance and System Audits

Definition

Performance audit - Evaluation to determine the accuracy of the total measurement system or components thereof

System audit - Evaluation to determine the proper selection and use of the measurement system, or components thereof

<u>External audits</u>

USATHAMA	EPA-CLP	COMMENTS
Reviewer: USATHAMA Analytical Branch	NPO Project Office Regional personnel EMSL/LV personnel CLP SMO NEIC	No substantial differences If Performance Evaluation samples were a required part of the USATHAMA plan, audits could be done simultaneously.
Frequency: After review of the project QC plan After initiation of analyses Other visits as deemed necessary	After first performance evaluation samples are completed Yearly Repeat site visit as needed	
Documentation: Checklist for laboratory adherence	Performance Evaluation sample score sheets Trend analysis Laboratory evaluation checklists	
Circulation of Audit Report: USATHAMA Project Officer Contractor Project Manager Analytical Task Manager Contractor QAC USATHAMA Analytical Branch		
Corrective action. Serious deficiencies are reported to the Contracting Officer at Procurement for action	Specified in contract under each QC section Evaluated by PO who may initiate a site visit, full data audit, or analysis of a second PE sample Laboratory may be placed on temporary hold Formulate recovery plan and SHOW CAUSE NOTICE	

12 Performance and System Audits (cont)

USATHAMA	EPA-CLP	COMMENTS
<u>Internal audits</u>		
Reviewer Project QC staff Frequency Not specified	Should be periodically conducted to evaluate the functioning of the QA SOP and involves an independent check of the laboratory analysts to ensure that procedures are being followed	
Documentation: Verification of maintenance of standards procedures, records, etc. Verification of actual practice vs. written procedures Verification of QA records and results of QC sample analyses Audit findings must be in a bound logbook		
Circulation of Audit Report: Project Manager Analytical Task Leader USATHAMA		

13 Preventive Maintenance

USATHAMA	EPA-CLP	COMMENTS
Schedule: Must maintain a calibration and maintenance schedule for each instrument as recommended by the manufacturer Physical or electronic measurements or calibrations must be traceable to NBS Supplies: An adequate supply of critical spare parts must be maintained Documentation: Reports and records must be available for inspection	Not specifically stated in contract, however, the following items are included in the laboratory <u>audit</u> checklist: service maintenance in-house replacement parts preventative maintenance permanent service record logbook instrument modifications	No difference

14. Specific Routine Procedures Used to Assess Data Precisision, Accuracy, and Completeness

USATHAMA	EPA CLP	COMMENTS
Software provided to assess precision and accuracy during certification	Contract specifies equations to evaluate precision and accuracy of matrix and surrogate spikes	USATHAMA procedure is superior and should be implemented if possible in CLP.
USATHAMA maintains a data management system which automates the statistical analyses of the data	Data is manually entered or copied from a floppy diskette into the EMSL/LV data-base for more extensive statistical review	

15. Corrective Action

USATHAMA	EPA-CLP	COMMENTS
Personnel responsible for initiating action:		No difference
Analyst	Analyst	
QAC		
Analytical Task Manager	Project Officer	
Project Manager		
Action:		
Immediate		
Repairing equipment	Specified in contract under each QC section	
Making a new standard		
Long term		
Staff training	Evaluated by PO	
Rescheduling	Laboratory may be placed on temporary hold	
Replacing vendors		
Revising of QA system	Formulate recovery plan	
Personnel replacement		
Documentation:		
Required	SHOW CAUSE NOTICE	

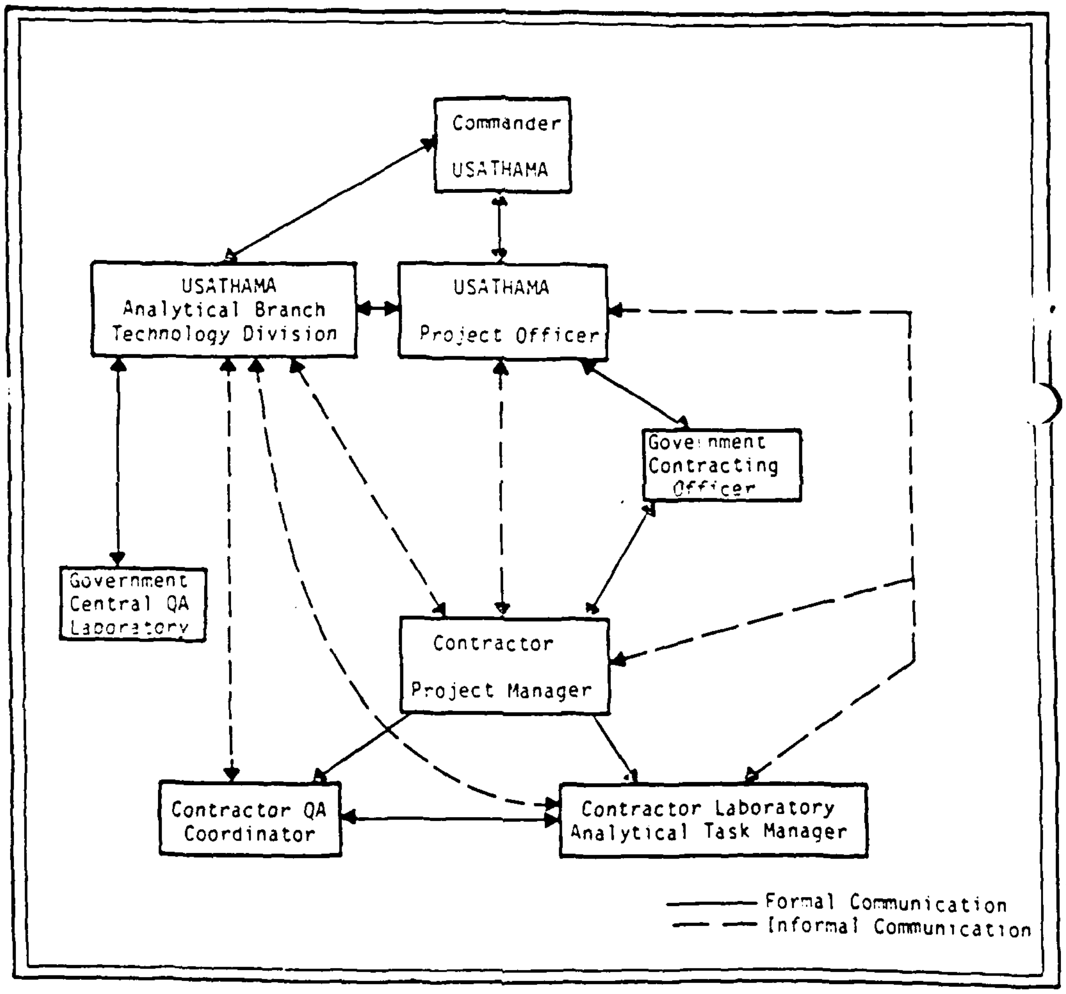
16. Quality Assurance Reports to Management

USATHAMA	EPA-CLP	COMMENTS
Precertification and certification performance data package IRDMS submissions Audit reports Control charts - provided to USATHAMA Project Officer weekly Final Project QC Data Report	Performance evaluation data package Data package submission Audit reports Quarterly Blind Performance Evaluation samples	QA report differences reflect the differences in other aspects of the plans, such as control charts, and Performance Evaluation samples. The advantages of each could be used.

Appendix A

LINES OF COMMUNICATION FOR USATHAMA IR PROJECTS
(USATHAMA QA PROGRAM, 2ND EDITION, MARCH, 1987)

Figure 2-1. Lines of Communication for USATHAMA IR Projects

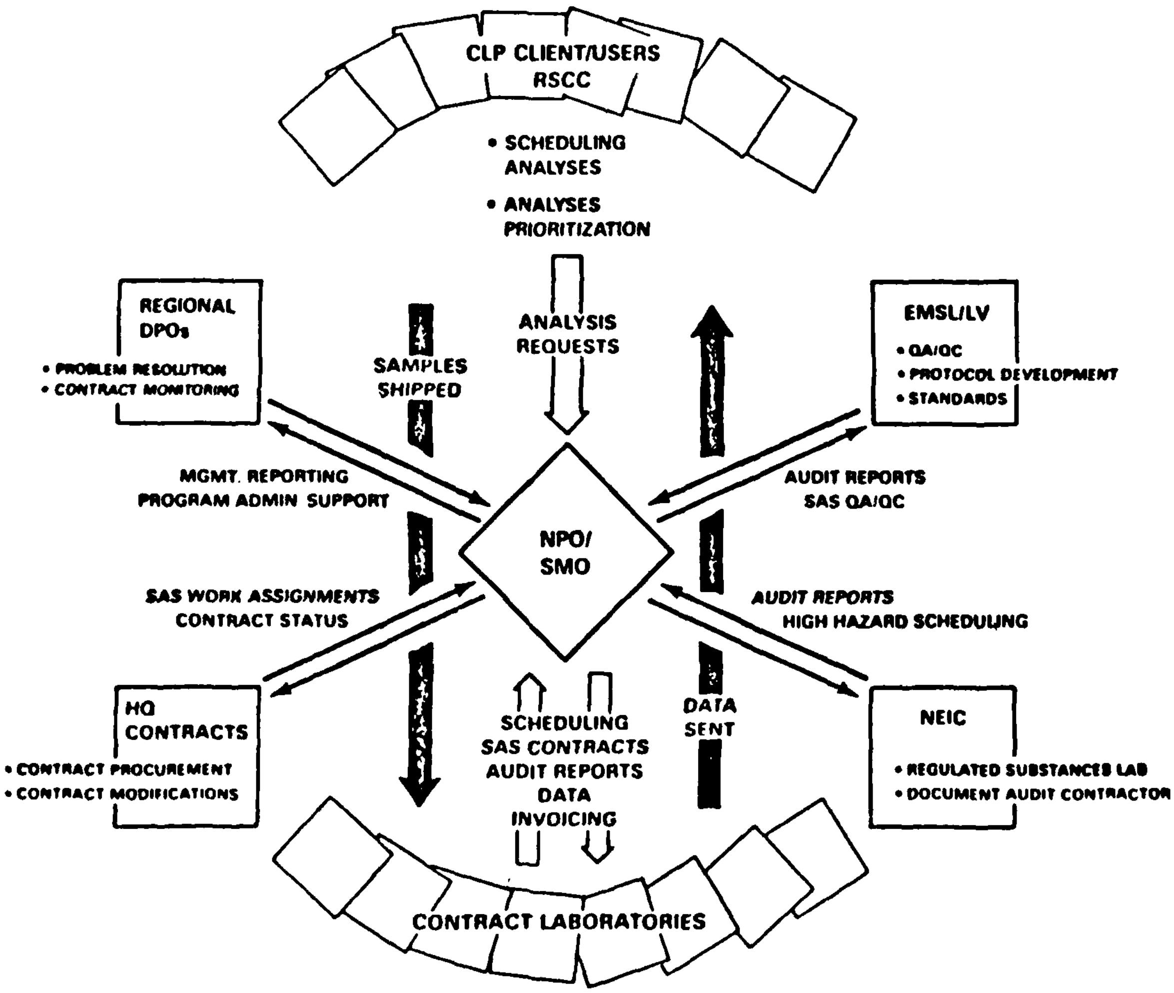


Appendix B

INTERRELATIONSHIP OF PROGRAM PRINCIPALS

(USER'S GUIDE TO THE CONTRACT LABORATORY PROGRAM, OCTOBER, 1984)

INTERRELATIONSHIP OF PROGRAM PRINCIPALS



Appendix C

CONTAINERS, PRESERVATION, STORAGE, AND HOLDING TIMES
AND
SAMPLE CONTAINER CLEANING PROCEDURES

(USATHAMA QA PROGRAM, 2ND EDITION, MARCH, 1987)

Table H-1. Containers, Preservation, Storage, and Holding Times[a]

Parameter	Container[b] Water	Soil	Preservative[c,d] Water	Soil	Maximum Holding Time for all Matrices[e]
INORGANIC TESTS					
Acidity	P	G	Cool, 4°C	Cool, 4°C	14 days
Alkalinity	P	G	Cool, 4°C	Cool, 4°C	14 days
Ammonia	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Asbestos	P	G	Cool, 4°C	Cool, 4°C	48 hours[f]
Bicarbonate	P	G	None Required	None Required	Analyze Immediately
Biochemical Oxygen Demand (BOD) and Carbonaceous BOD	P	G	Cool, 4°C	Cool, 4°C	48 hours
Bromide	P	G	None Required	None Required	28 days
Carbonate	P	G	None Required	None Required	Analyze Immediately
Chemical Oxygen Demand (COD)	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Chloride	P	G	None Required	None Required	28 days
Chlorine, Total Residual	P	N/A	None Required	N/A	Analyze Immediately
Color	P	N/A	Cool, 4°C	N/A	48 hours

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Cyanide, Total and Amenable to Chlorination	P	G	Cool, 4°C NaOH to pH >12 0.6 g Ascorbic Acid[g]	Cool, 4°C	14 days [h]
Dissolved Oxygen					
Probe	G Bottle and Top	N/A	None Required	N/A	Analyze Immediately
Winkler	G Bottle and Top	N/A	Fix On Site *Store in Dark*	N/A	8 hours
Fluoride	P	G	None Required	None Required	28 days
Hardness	P	N/A	HNO_3 or H_2SO_4 to pH<2	N/A	6 months
Hydrazine	P	G	If not analyzed immediately, collect under acid. Add 90 ml of sample to 10 ml HCl.	Cool, 4°C	7 days
Iodide	P	G	Cool, 4°C	Cool, 4°C	24 hours
Iodine	P	G	None Required	None Required	Analyze Immediately
Kjeldahl and Organic Nitrogen	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Metals[i]					
Chromium VI	P	G	Cool, 4°C	Cool, 4°C	24 hours
Mercury	P	G	HNO_3 to pH <2	Cool, 4°C	28 days
Others	P	G	HNO_3 to pH <2	Cool, 4°C	6 months
Nitrate	P	G	Cool, 4°C	Cool, 4°C	48 hours
Nitrate plus Nitrite	P	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Nitrite	P	G	Cool, 4°C	Cool, 4°C	48 hours
Oil and Grease	G	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Orthophosphate	P	G	Filter Immediately Cool, 4°C	Cool, 4°C	48 hours
pH	P	G	None Required	None Required	Analyze Immediately
Phenols	G	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Phosphorous, Elemental	G	G	Cool, 4°C	Cool, 4°C	48 hours
Phosphorous, Total	P,G	G	Cool, 4°C H_2SO_4 to pH <2	Cool, 4°C	28 days
Silica, Dissolved or Total	P	G	Cool, 4°C	Cool, 4°C	28 days

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Residue					
Filterable	P	N/A	Cool, $4^{o}C$	N/A	7 days
Settleable	P	N/A	Cool, $4^{o}C$	N/A	48 hours
Nonfilterable (TSS)	P	N/A	Cool, $4^{o}C$	N/A	7 days
Total	P	N/A	Cool, $4^{o}C$	N/A	7 days
Volatile	P	N/A	Cool, $4^{o}C$	N/A	7 days
Specific Conductance	P	G	Cool, $4^{o}C$	Cool, $4^{o}C$	28 days
Sulfate	P	G	Cool, $4^{o}C$	Cool, $4^{o}C$	28 days
Sulfide	P	G	Cool, $4^{o}C$ Add Zinc Acetate plus NaOH to pH >9	Cool, $4^{o}C$	7 days
Sulfite	P	G	None Required	None Required	Analyze Immediately
Surfactants	P	G	Cool, $4^{o}C$	Cool, $4^{o}C$	48 hours
Temperature	P	G	None Required	None Required	Analyze Immediately
Turbidity	P	N/A	Cool, $4^{o}C$	N/A	48 hours
ORGANIC TESTS[j]					
Acrolein and Acrylonitrile	S	S	Cool, $4^{o}C$ 0.008% $Na_2S_2O_3$[g] Adjust pH to 4-5[k]	Cool, $4^{o}C$	14 days[k]

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Benzidines[l]	G	G	Cool, $4^{o}C$[m] 0.008% $Na_2S_2O_3$[g] pH 2-7	Cool, $4^{o}C$	7 days until extraction[n]
Chlorinated Hydrocarbons[l]	G	G	Cool, $4^{o}C$	Cool, $4^{o}C$	7 days until extraction 40 days after extraction
Haloethers[l]	G	G	Cool, $4^{o}C$ 0.008% $Na_2S_2O_3$[g]	Cool, $4^{o}C$	7 days until extraction 40 days after extraction
Nitroaromatics and Isophorone[l]	G	G	Cool, $4^{o}C$ Store in Dark	Cool, $4^{o}C$ Store in Dark	7 days until extraction 40 days after extraction
Nitrosamines[l,o]	G	G	Cool, $4^{o}C$ Store in Dark 0.008% $Na_2S_2O_3$[g]	Cool, $4^{o}C$ Store in Dark	7 days until extraction 40 days after extraction
PCBs	G	G	Cool, $4^{o}C$	Cool, $4^{o}C$	7 days until extraction 40 days after extraction
Pesticides[l]	G	G	Cool, $4^{o}C$ pH 5-9[p]	Cool, $4^{o}C$	7 days until extraction 40 days after extraction
Phenols[l]	G	G	Cool, $4^{o}C$ 0.008% $Na_2S_2O_3$[g]	Cool, $4^{o}C$	7 days until extraction 40 days after extraction
Phthalate Esters[l]	G	G	Cool, $4^{o}C$	Cool, $4^{o}C$	7 days until extraction 40 days after extraction

Table H-1. (Cont'd.)

Parameter	Container[b] Water	Container[b] Soil	Preservative[c,d] Water	Preservative[c,d] Soil	Maximum Holding Time for all Matrices[e]
Polynuclear Aromatic Hydrocarbons[f]	G	G	Cool, 4°C 0.008% $Na_2S_2O_3$[g] Store in Dark	Cool, 4°C Store in Dark	7 days until extraction 40 days after extraction
Purgeable Aromatic Hydrocarbons	S	S	Cool, 4°C 0.008% $Na_2S_2O_3$[g] HCl to pH <2[g]	Cool, 4°C	14 days[g]
Purgeable Halocarbons	S	S	Cool, 4°C 0.008% $Na_2S_2O_3$[g]	Cool, 4°C	14 days
TCDD[f]	G	G	Cool, 4°C 0.008% Na_2SO_3[g]	Cool, 4°C	7 days until extraction 40 days after extraction
Total Organic Carbon	G	G	Cool, 4°C HCl or H_2SO_4 to pH <2	Cool, 4°C	28 days
Total Organic Halogen	G	G	Cool, 4°C 1 ml of 0.1 M sodium sulfite	Cool, 4°C	7 days

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Analytes not listed should be preserved at 4°C and held not longer than 7 days.

[a]Preservatives and holding times are from Federal Register, Vol. 49, No. 209, Friday, October 26, 1984, Page 43260 and Characterization of Hazardous Waste Sites: A Methods Manual -- Volume II, Sampling Methods, Second Edition, EPA-600/4-84-076. Container requirements are consistent with these references.

[b]P = Polyethylene
G = Amber Glass with Teflon-lined cap
S = Glass Vial with Teflon-lined septum cap

[c]Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot *should be preserved at the time of collection*. When *use of an automatic sampler makes* it impossible to preserve each aliquot, samples may be preserved by maintaining at $4^{0}C$ until compositing and sample splitting is completed.

[d]When any sample is to be shipped by common carrier or sent through the U.S. Mail, it must comply with the Department of Transportation *Hazardous Materials Regulations (49 CFR Part 172). The person offering such* material for transportation is responsible for ensuring such compliance. For the preservation requirements in this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation, has *determined* that the Hazardous Materials Regulations do not *apply to the following* materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.3 or less).

[e]Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples *may be held before analysis and still be considered valid*.

Some samples may not be stable for the maximum time period given in the table. A laboratory is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample integrity.

[f]If samples cannot be filtered within 48 hours, add 1 ml of a 2.71% solution of mercuric chloride to inhibit bacterial growth.

[g]Should only be used in the presence of residual chlorine.

[h]Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[i]For dissolved metals, filter immediately on site before adding preservative.

[j]Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[k]The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.

[l]When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times must be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and 40 days after extraction. Exceptions to this optimal preservation and holding time procedure are noted in footnotes g, m, and n.

[m]If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[n]Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[o]For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

[p]The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[q]Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

APPENDIX G

SAMPLE CONTAINER CLEANING PROCEDURES

To ensure the integrity of aqueous and solid samples, steps must be taken to minimize contamination from the containers in which they are stored. If the analyte(s) to be determined are organic in nature, the container should be made of amber glass. If the analyte(s) are inorganic, the container should be polyethylene. When both organic and inorganic substances are expected to be present, separate samples should be taken. New sample bottles must be cleaned according to the procedure presented below; reuse of sample containers is expressly prohibited. Commercially cleaned containers may be utilized if cleaning procedures comply with those provided in this appendix and prior USATHAMA approval is obtained. The procedures for cleaning the glass and polyethylene containers and their caps are as follows:

- Polyethylene Bottles and Polyethylene Caps

  (1) Rinse bottles and lids with 5% sodium hydroxide.

  (2) Rinse with deionized water.

  (3) Rinse with 5% Ultrex (or equivalent) nitric acid in deionized water.

  (4) Rinse with deionized water.

  (5) Drain and air dry.

- Amber-Glass Bottles or 40-ml Vials

  (1) Scrub and wash bottles in detergent.

  (2) Rinse with copious amounts of distilled water.

  (3) Rinse with acetone.

  (4) Rinse with methylene chloride (Nanograde or equivalent).

  (5) Rinse with hexane (Nanograde or equivalent).

  (6) Air dry.

  (7) Heat to 200°C.

  (8) Allow to cool.

  (9) Cap with clean caps with Teflon liners.

- Bottle Caps

  (1) Remove paper liners from caps.

  (2) Wash with detergent.

  (3) Rinse with distilled water.

  (4) Dry at $40^{o}C$.

- Teflon Liners (avoid contact with fingers)

  (1) Wash with detergent.

  (2) Rinse with distilled water.

  (3) Rinse with acetone.

  (4) Rinse with hexane (Nanograde or equivalent).

  (5) Air dry.

  (6) Place liners in cleaned caps.

  (7) Heat to $40^{o}C$ for 2 hours.

  (8) Allow to cool.

  (9) Use to cap cleaned bottles.

Appendix D

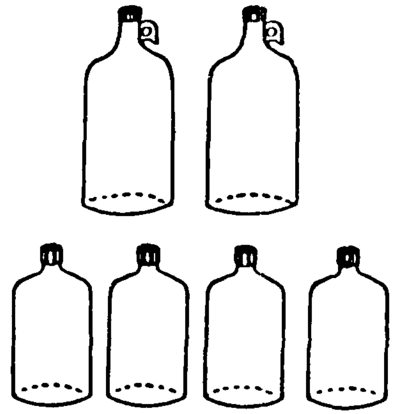
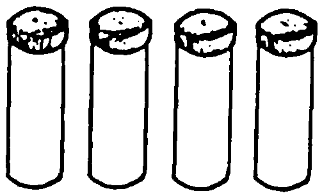
ORGANIC SAMPLE COLLECTION REQUIREMENTS
AND
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

(USER'S GUIDE TO THE CONTRACT LABORATORY PROGRAM, OCTOBER, 1984)

AND

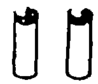
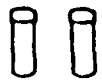
(CONTRACT LABORATORY PROGRAM STATEMENT OF WORK
FOR INORGANIC ANALYSIS, OCTOBER, 1986 REV.)

ORGANIC SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW LEVEL)	1 GALLON		2 X 80-OZ. AMBER GLASS BOTTLES OR 4 X 1-LITER AMBER GLASS BOTTLES
EXTRACTABLE ANALYSIS (MEDIUM LEVEL*)	1 GALLON		4 X 32-OZ. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	80 ML		2 X 40-ML GLASS VIALS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

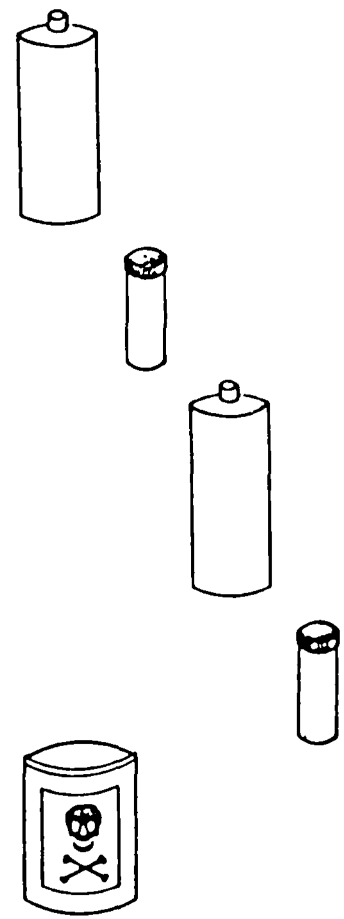
ORGANIC SAMPLE COLLECTION REQUIREMENTS

SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.		1 X 8-OZ. WIDE-MOUTH GLASS JAR
			OR
			2 X 4-OZ. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	240 ML		2 X 120-ML WIDE-MOUTH GLASS VIALS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

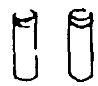
INORGANIC SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED VOLUME	CONTAINER TYPE
METALS ANALYSIS (LOW LEVEL)	1 LITER	1 X 1-LITER POLYETHYLENE BOTTLE
METALS ANALYSIS (MEDIUM LEVEL*)	16 OZ.	1 X 16-OZ. WIDE-MOUTH GLASS JAR
CYANIDE (CN^-) ANALYSIS (LOW LEVEL)	1 LITER	1 X 1-LITER POLYETHYLENE BOTTLE
CYANIDE (CN^-) ANALYSIS (MEDIUM LEVEL*)	16 OZ.	1 X 16-OZ. WIDE-MOUTH GLASS JAR



*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

INORGANIC SAMPLE COLLECTION REQUIREMENTS

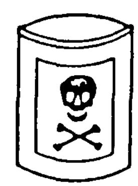
SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
METALS AND CYANIDE (CN^-) ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.		1 X 8-OZ. WIDE-MOUTH GLASS JAR
			OR
			2 X 4-OZ. WIDE-MOUTH GLASS JARS

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

HIGH HAZARD SAMPLE COLLECTION REQUIREMENTS

	REQUIRED VOLUME		CONTAINER TYPE
LIQUID SAMPLES			
ORGANIC AND INORGANIC ANALYSIS	6 OZ.		1 X 8-OZ. WIDE-MOUTH GLASS JAR
SOLID SAMPLES			
ORGANIC AND INORGANIC ANALYSIS	6 OZ.		1 X 8-OZ. WIDE-MOUTH GLASS JAR

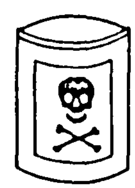
*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT



DIOXIN SAMPLE COLLECTION REQUIREMENTS

SOIL/SEDIMENT <u>SAMPLES</u>	REQUIRED <u>VOLUME</u>		<u>CONTAINER TYPE</u>
2.3.7.8-TCDD (DIOXIN) ANALYSIS	4 OZ.		1 X 4-OZ. WIDE-MOUTH GLASS JAR
			OR
			1 X 8-OZ. WIDE-MOUTH GLASS JAR

*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT



Required Containers, Preservation Techniques, and Holding Times

Measurement Table/Parameter		Container[1]	Preservative[2,3]	Maximum Holding Time For Water Samples[4]
IB	Inorganic Tests			
23-24,	Cyanide, total and amenable to chlori nation	P,G	0.6g ascorbic acid[5] NaOH to pH >12 Cool, 4°C	14 days [6]
	Metals[7]			
35,	Mercury	P,G	HNO_3 to pH <2	26 days
IB 3, 5-8, 11, 13, 14, 19, 20, 22, 26 29, 30, 32-34, 36, 37, 45, 47, 51 52, 58, 59, 60, 62, 63, 70-72, 74 75,	Metals, except above	P,G	HNO_3 to pH <2	6 months

See following page for notes.

Notes

1. Polyethylene (P) or Glass (G).

2. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4°C ($\pm$5°C) until compositing the sample splitting is completed.

3. When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCL) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H2SO4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater; and Sodium hydroxide (NaOH) in water solution at concentration of 0.080% by weight or less (pH about 12.30 or less).

4. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid.

5. Should only be used in the presence of residual chlorine.

6. Maximum recommended holding time is less when sulfide is present. Optionally, all samples may be tested with lead acetate paper before the pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

7. Samples should be filtered immediately on-site before adding preservative for dissolved metals.

DEVELOPMENT OF THE QUALITY ASSURANCE TASK FORCE

Experts from many scientific and regulatory agencies met in Las Vegas, Nevada on February 18-20, 1987 to discuss Quality Assurance/ Quality Control. The objectives of the group were to exchange ideas, share resources and technology, explore creative approaches, address key issues, and hopefully develop a unified plan for assuring quality data. A list of the attendees is included in Appendix A. The participants were divided into seven working groups so that the following topics could be discussed in greater detail:

Group 1 - Quality Assurance Management and Data Quality Objectives

Group 2 - On-Site Auditing, Data Review, and Evaluation

Group 3 - Performance Evaluation and Reference Material

Group 4 - Method Validation and Equivalency

Group 5 - Sample Management, Holding Times and Chain of Custody

Group 6 - Statistics and Chemometrics

Group 7 - Documentation and Data Communication

In order to facillitate the continuing exchange of ideas and resources, a proposal was submitted to the group for the formation of a Quality Assurance Task Force. The Quality Assurance Task Force would promote the continual development of a unified approach to QA/QC.

A summary of the findings of each of the working groups is presented below:

GROUP 1 - QUALITY ASSURANCE MANAGEMENT AND DATA QUALITY OBJECTIVES

Group 1 emphasized that the Army and EPA should define what types of data are needed, note comparisons and differences in data packages, and address methods to meet data requirements. Follow-up meetings on these and other differences should be held. The decision-making personnel in each agency should be identified and be responsible for establishing equivalency. The results should then be communicated to all ten regions.

GROUP 2 - ON-SITE AUDITING, DATA REVIEW, AND EVALUATION

Group 2 concluded that there was no consistency among the different agencies concerning precertification and certification. EPA's CLP program requires prospective labs to demonstrate, at their

own cost, administrative and technical capabilities before the contract is awarded. USATHAMA awards contracts, through a RFP process, on the basis of a written proposal and the history of the laboratory. Therefore, demonstration of technical profiency is paid for by USATHAMA. USDA requires a performance evaluation sample for accreditation. Failure on the performance evaluation sample necessitates that the laboratory must wait for six months before reapplication. The EPA CLP considers the postaward performance evaluation samples to be a major topic for the on-site laboratory evaluation, unlike the USATHAMA, the Navy, or the USDA. The group agreed that the on-site evaluation checklist was fairly uniform, but that the frequency of the audits and the level of corrective action applied varied with the agency. The EPA CLP was the only program which looked for serious problems by reconstructing final results from the original raw data during the audit. Some members of the group expressed interest in on-site auditing of the field sampling process. Also, the group proposed that the issues and benefits of agencies sharing laboratory performance information should be addressed.

Group 2 found that even though the time frame allowed for the review of data ranged from one week to three months, the procedures were generally the same. All reviewers looked for outlying data, controls, suspicious calibrations, etc. The group did emphasize that they did not feel that data was over-reviewed. Even though data was reviewed in the same manner, the application of the data depended on the end user and could vary widely. Differences in reviews depended on the auditor's function in the overall project scheme. Any data that was involved in litigation and chain of custody would take longer to review. The group decided that the audit could be facillitated by computerized data scans, organized standard data packages, and easier access to lab personnel and sampling information.

GROUP 3 - PERFORMANCE EVALUATION AND REFERENCE MATERIAL

Group 3 began their working session by identifying several issues to discuss. The group felt that the level of effort required to "certify a material" needed to be defined and that increased traceability of materials to NBS would be desirable. Purity documentation of standard materials should include one identity and two purity analyses and only designated labs should be allowed to certify materials. Also reference materials should have a certificate of analysis with dates of preparation and expiration, methods used, and any other pertinent data. Interagency cooperation in making and using batches of reference materials would increase the amount of funding available to make and certify Standard Reference Materials from NBS. The group felt that an interagency work statement was needed to plan a feasibility study on soil reference material preparation and analysis.

Group 3 was then divided into three subgroups: definitions, performance evaluation materials, and reference materials. The definitions subgroup defined reference materials as a general term for characterized substances used for the following three functions: instrument calibration, intralaboratory QC, and interlaboratory performance evaluation.

1. The purpose of calibration is to establish the relationship between instrument response and concentration. Calibration is accomplished by using calibration standards which are well characterized as to purity, stability, and concentration.

2. The purpose of intralaboratory quality control is to provide an assessment of data quality within a laboratory. This information is developed in part by daily analysis of a laboratory check sample. These check samples can be prepared by the laboratory or obtained from an external source. In addition, the laboratory should periodically analyze externally supplied check materials of known composition, such as EPA's QC samples and the SRM's from NBS.

3. The purpose of performance evaluation is to provide an assessment of the comparability of analytical results between laboratories. Performance evaluation materials (PEM's) are periodically supplied to the laboratory as unknowns by the sponsoring agency.

   PEM's may be derived from natural matrix materials or synthetically prepared. The evaluation of laboratories using PEM's may be based on comparison with known values or by comparison of individual results against the average performance.

The subgroup on performance evaluation materials discussed the need for solid organic performance evaluation samples, acknowledging that nonvolatile evaluation samples are relatively easy to prepare, but that studies were needed for preparation of volatiles in solids. A need for performance evaluation samples for explosives in soil was expressed. The subgroup decided that natural contaminated matrices were preferable to spiked matrices if possible. The frequency and character of the performance evaluation samples was discussed, as well as the selection of analytes, matrices, and concentrations. The necessity of keeping evaluation samples as blind as possible was recognized, with the minimum frequency being one blind performance evaluation sample per lot of samples. However, studies should be made of laboratory operations to determine the optimum frequency, with a combination of control charts and performance evaluation samples being the best approach. The subgroup decided that the performance evaluation samples should be at multiple levels of concentration

(taking into account the method), and should contain well characterized analytes of low intrinsic variability. Both easy and difficult analytes should be included, as well as those that are easily confused. Performance evaluation results should be purged of proprietary information and shared between the different agencies. Matrices needed to be representative of the sites under study. Selection of six to ten representative types of soil was suggested. The need for a catalog of sources of environmental performance evaluation samples was expressed. The subgroup also discussed the evaluation of the performance of laboratories from evaluation sample data. The use of the results and the formation of data quality objectives should be consistent with the end use of the data such as risk assessment and legal considerations. Education of the end users of the data was suggested because of misconceptions concerning the width of windows for evaluating the results. Participants also reaffirmed that performance evaluation sample results only comprised one portion of the laboratory evaluation and that results would be of marginal value if criteria limits were set near method detection limits. Comments were made that dilution errors were a problem with high level performance evaluation samples and outliers on the high and low sides needed to be handled differently.

GROUP 4 - METHOD VALIDATION AND EQUIVALENCY

The first concern of Group 4 was to define method validation in the following manner:

> Method Validation is a process starting with definition of method analytes and sample matrices followed by identification of suitable existing methods for conducting the analysis. A method is selected, optimized and validated in a single laboratory study which must include determination of method detection limit, precision and bias in a range of matrices of interest and also include ruggedness testing and writing a complete method protocol. Following a rigorous single lab study, the method undergoes collaborative testing by a minimum of six to eight laboratories. The method is considered to be validated if the written protocol can be followed by the participating laboratories, the method is suitable for the tested matrices, and if the precision and accuracy of the collaborative results are within the limits set in the Data Quality Objectives.

The group also recognized that method validation is a separate process from certification of a laboratory to perform a method. In validating a method, the natural environmental matrix was preferred if the process of locating and characterizing the matrix did not exhaust the available resources. Fortified natural matrices could be used as well as a totally synthetic matrix. Problems associated with obtaining laboratories to participate in interlaboratory studies was discussed. The group felt that competition among the laboratories might make them commit to a validation effort, but there were no guarantees that the data would be delivered in a timely manner.

Guidelines for conducting dynamic validation were developed by the group. It should only be used when more than 20 laboratories are participating, the method must be based on a previously tested method for which there is a high degree of confidence that its performance will meet or exceed program requirements, and the method must include a strong quality control program.

Group 4 recommended that the EPA Superfund staff adopt a policy which would allow other federal agencies to demonstrate equivalency of their methods to Superfund methods. Problems mentioned which could result from adopting such a policy were different reporting requirements and proliferation of equivalent methods which would make data review more difficult.

The group recommended that other federal agencies be invited to attend future caucuses and that the QA workshop should be reconvened in one year or less.

GROUP 5 - SAMPLE MANAGEMENT, HOLDING TIMES AND CHAIN OF CUSTODY

Group 5 recognized that a uniform sample definition would be desirable, but may not be possible. However, sample terms must be defined and mutually understood by all agencies involved. QA procedures for field sampling, field logs, and chain-of-custody documentation should be uniform. Total compliance in maintaining chain-of-custody could be very difficult with more automated analyses. More stringent QA concerning field sampling is needed since this represents a huge source of error. Some estimate of this error would be desirable. The group felt that information on the validity of holding times would be desirable in unifying specifications among the various agencies. Requirements for reanalysis were different among the agencies if checking of data revealed that analyses were inappropriately performed. A centralized database as a means of obtaining summary information on laboratories such as current standing, date of most recent audit, and date of most recent performance evaluation sample analysis would be advantageous to all agencies concerned.

GROUP 6 - STATISTICS AND CHEMOMETRICS

Group 6 raised five issues for discussion. The first issue was detection limits. The group established that the detection limit represents a concentration where decisions about presence or absence are made and that the quantitation (reporting) limit is at some concentration above the detection limit. The detection limit is highly dependent on the individual characteristics of the apparatus, analyst, method, etc. Data was shown to suggest that EPA's MDL and THAMA's Hubaux and Vos estimates from some data sets show a maximum difference

in ratio of 1.5. The group concluded that the two procedures were not as different as thought at first, and expressed a need for more information on how to set limits for multi-analyte methods, using surrogates to evaluate matrix variations, more comparative evaluations of the different methods of estimating detection limits, more education on the variables which are included in the detection limit estimates, and a determination of the most effective way to specify concentration limits and evaluate inherent risks.

The second issue raised was chemometrics. During this discussion, the following needs were expressed: investigation of applications of composite sampling techniques to environmental monitoring, estimation of variablility due to sampling, improved laboratory subsampling protocols, and more nested experimental programs to provide objective estimates based on real samples of the following: field sampling variability, lab subsampling/preparation, and analytical variablility.

The third issue that the group discussed was the development of performance evaluation sample criteria. Double-blind performance evaluation sample submission was considered ideal when feasible at a frequency consistent with the needs of the program and cost benefit goals. Ultimately the group wanted to see capability limits established for the performance evaluation standards for various methods and for different types of evaluation materials. In establishing these criteria, the problem of editing data to exclude true "outliers" without unduly truncating data sets was recognized. The question was also raised about the effect of a large number of outliers in a data set upon future repeatability. A suggestion was made to use the Biweight Robust Estimation Procedure (JASA, June 1982) which provides the basis for USEPA performance evaluation criteria limits for water analysis. Some out-of-control data could be discarded if the frequency of performance evaluation checks is increased.

The group labeled the fourth issue as design/analysis comparability. Essentially, improved communication between lab personnel and field samplers was a necessity in order to carefully formulate all sampling protocols and analysis in advance so that all statistical computations would be compatible with the actual performance of the experiments. Any uncontrolled variables in the procedures also needed to be noted to aid in the design process as well as final use of the data (qualitative vs. quantitative).

Concerning the last issue, validation of reference materials, the group required detailed procedures to establish homogeneity and stability.

GROUP 7 - DOCUMENTATION AND DATA COMMUNICATION

Group 7 agreed that the quality and quantity of the documentation required for a program varies depending on the end use of the data from the most intensive documentation necessary for litigation purposes to minimum documentation only needed for characterization/information gathering to make rapid decisions. The group stated that sampling and analytical contracts should require the level of documentation necessary for the program's purposes with the long term goal that the various agencies would reach some consensus on what documentation should be required. Meeting this goal would solve such problems as agreement of EPA and DOD on documentation when both agencies are involved in sites on DOD facilities and alleviate the frustration of contractor labs who are required to follow different documentation and reporting requirements depending on the agency. The group questioned whether software systems could be developed and implemented to process some of the quality control elements common to all the agencies and provide some documentation. USATHAMA followed up this discussion with a presentation of their computer software.

DISTRIBUTION LIST

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1. Central Research Library, 4500-N, MS 286
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13. Laboratory Records, 4500-N, MS 285
14. Mr. J. A. Lenhard, DOE/ORO

15-16. S. K. Holladay, 4500-S, MS 120

17-26. M. P. Maskarinec, 4500-S, MS 120

27. M. Miller, K-1004-C, MS 440
28. ORNL Patent Office, 4500-N, MS 258

External

29-40. Defense Technical Information Center, ATTN: DTIC-DDA, Cameron Station, Alexandria, VA 22314.

41-42. Commander, U.S. Army Toxic and Hazardous Materials Agency, ATTN: AMXTH-CO-P, Aberdeen Proving Ground, MD 21010-5401.

43-44. Defense Logistics Studies Information Exchange, U.S. Army Logistics Management Center, Fort Lee, VA 23801.

45-49. Mary Ann Ryan, USATHAMA, AMXTH-TE-A, Bldg. E-4585, Aberdeen Proving Ground, MD 21010-5401.

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71. Mike Homsher, Lockheed EMSCO, P.O. Box 15027, Las Vegas, NV 89114.
72. Chuck Hoover, Lockheed EMSCO, P.O. Box 15027, Las Vegas, NV 89114.
73. William Horwitz, FDA, Center for Food, Safety and Applied Nutrition, HFF-7, Washington, DC 20204.
74. Peter Isaacson, Sample Management Office, P.O. Box 918, Alexandria, VA 22313.
75. Tom Jenkins, USACRREL, ATTN: CRREL-RC, Hanover, NH 03775.
76. Captain Philip Jung, USAF, OEHL/TF, Brooks Air Force Base, TX 78235-5000.
77. D. A. Kane, USAF, HQ USAF/LEEV, Bolling Air Force Base, Washington, DC 20332.
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84. Paul Marsden, S-Cubed, P.O. Box 1620, La Jolla, CA 92038.
85. Bill Maurits, Product Assurance Direct, AMSMC-QAO-C(A), Aberdeen Proving Ground, MD 21010.
86. Willie May, National Bureau of Standards, Gaithersburg, MD 20899.
87. Ron Mitchum, USEPA EMSL/LV, P.O. Box 15027, Las Vegas, NV 89114.
88. John Moore, USEPA EMSL/LV, P.O. Box 15027, Las Vegas, NV 89114.
89. J. Gareth Pearson, USEPA/LV, P.O. Box 15027, Las Vegas, NV 89114.

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